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On-surface formation of cumulene by dehalogenative homocoupling of alkenyl gem-dibromides

Qiang Sun, Bay V. Tran, Liangliang Cai, Honghong Ma, Xin Yu, Chunxue Yuan, Meike Stöhr*, and Wei Xu*

Abstract: The on-surface activation of carbon-halogen groups represents an efficient route to produce radicals for constructing various hydrocarbons as well as carbon nanostructures. To the best of our knowledge, the so far employed halide precursors have only one halogen attached to a carbon atom. It is thus of particular interest to study the effect of attaching more than one halogen atom to a carbon atom with the aim of producing multiple unpaired electrons. Herein, by introducing an alkenyl gem-dibromide, we have successfully fabricated cumulene products on a Au(111) surface by dehalogenative homocoupling reactions. The reaction products and pathways were unambiguously characterized by the combination of high-resolution scanning tunneling microscopy (STM) and non-contact atomic force microscopy (nc-AFM) measurements together with state-of-the-art density functional theory (DFT) calculations. This study further supplements the database of on-surface synthesis strategies, and importantly, it provides a facile manner for incorporation of more complicated carbon scaffoldings into surface nanostructures.

The recently developed on-surface synthesis strategy has gained increasing attention due to its prospects in constructing - starting from organic monomers - covalently interlinked, extended nanostructures, which are of scientific and technological interest for future applications owing to their promising electronic properties and high mechanical stability.¹⁻⁶ In general, most of the on-surface reactions follow pathways different from their counterparts in solution because of the effects of the surface: the reactions are confined in two dimensions and the possible catalytic surface activity.⁷⁻¹⁴ Consequently, unexpected reactions have been surprisingly discovered in on-surface synthesis experiments,¹⁵⁻²² and thus, this strategy has opened up a way for the fabrication of a plethora of novel surface nanostructures which may be hardly obtained by traditional solution methods. Among others, the atomically precise synthesis of carbon nanostructures such as graphene nanoribbons²³⁻³⁰ and other hydrocarbons like alkanes, dienes and diynes has become a hot topic within the field of on-surface synthesis.³¹⁻³⁶ In particular, it has been demonstrated that

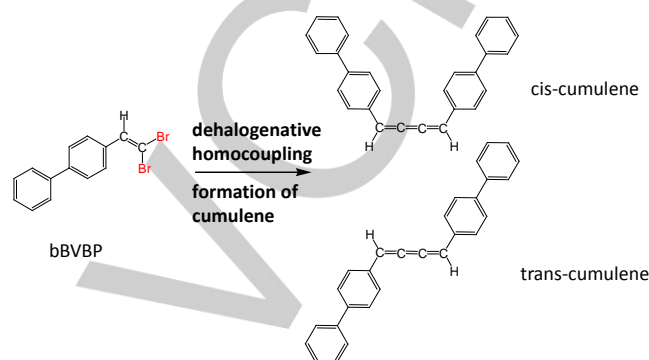


Figure 1. Schematic illustration showing the formation of cis- and trans-cumulene products through dehalogenative C-C homocoupling reactions of the alkenyl gem-dibromide molecular precursor bBVBP.

the thermally-induced dehalogenation of pre-defined C-X groups (X stands for halogens) provides an efficient route to produce radicals for subsequent C-C couplings on surfaces.^{14,24,27} Besides, some reactive intermediates, like arynes,³⁷ aromatic diradicals and a highly strained ten-membered diyne³⁸ have been artificially generated on surfaces by cleaving C-X bonds with the aid of STM manipulations and thereby, yielding radicals. However, to the best of our knowledge, the so far employed halide precursors have only one halogen atom attached to a carbon atom, which can merely produce one unpaired electron at the carbon site. Thus, it is of particular interest to investigate dehalogenative reactions of geminal dihalide precursors on surfaces, to explore and thereby, extend our fundamental understandings of on-surface C-C coupling reactions with two unpaired electrons.

Recently, we have investigated the dehalogenative homocoupling of alkenyl bromides on Cu(110).³⁹ In light of this previous study, we have designed and synthesized a molecular precursor functionalized with an alkenyl gem-dibromide group (4-(2,2-dibromovinyl)-1,1'-biphenyl, named bBVBP), as shown in Figure 1. Herein, by the combination of high-resolution STM and nc-AFM measurements, we investigate the debromination and the subsequent C-C homocoupling reactions of alkenyl gem-dibromides on a Au(111) surface. We unambiguously identify from the single-bond resolved nc-AFM images the formation of cis- and trans-cumulene products having three consecutive C-C double bonds as schematically shown in Figure 1. These findings are explained by successive debromination reactions followed by subsequent C-C homocouplings on the basis of our DFT-based transition-state search calculations. This study extends the database of on-surface dehalogenative C-C homocoupling reactions by introducing gem-dibromides. Importantly, it provides a facile manner to generate two unpaired electrons by a surface-assisted successive C-Br bond activation resulting in the formation of C-C double bonds (specifically, cumulenes in this case) based on C-C homocouplings. This represents a new strategy for incorporating

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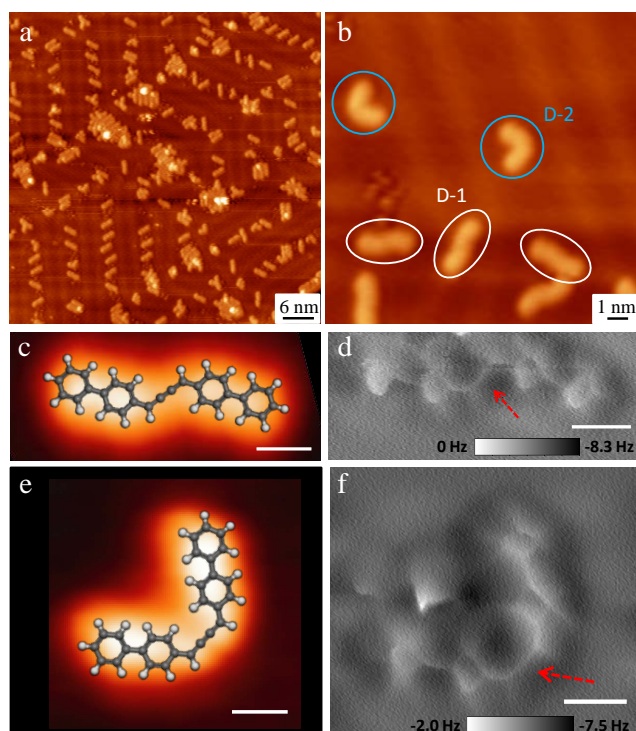


Figure 2. Formation of cumulene on Au(111). (a) Overview STM image showing the formation of discrete motifs after deposition of bBVBP molecules on Au(111) at room temperature. (b) Close-up STM image allowing to identify two typical dimer structures, labeled as D-1 and D-2, respectively. Scanning parameters: $V_t = -2.1$ V, $I_t = 20$ pA. (c,e) High-resolution STM images of the dimer structures D-1 and D-2, respectively, together with the equally scaled DFT relaxed models overlaid on top. (d,f) Corresponding frequency shift AFM images of the two dimer structures. The red arrows indicate the cumulene groups. Scale bars: 5 Å.

more complicated carbon scaffoldings into tailor-made surface nanostructures.

After deposition of bBVBP molecules on Au(111) held at room temperature, we observed the formation of discrete structural motifs as well as small aggregations on the surface as shown in the overview STM image in Figure 2a. A closer inspection of the STM image allows to identify that most of the molecules reside at the fcc regions or elbow sites of the herringbone reconstruction of the Au(111) surface. The two most abundant motifs observed are shown in Figure 2b: one displays a linear shape with two lobes (labeled D-1) and the other displays a curved shape with two lobes as well (labeled D-2). Based on the STM appearances and dimensions of the two lobes, we infer that both D-1 and D-2 are composed of two bBVBP molecules. According to our previous study,³⁹ such D-1 and D-2 dimers are speculated to be the products of dehalogenative C-C homocoupling reactions on the surface. However, the bond configurations within the dimer structures cannot be precisely determined based on the STM images. Note that we occasionally observe a trimer structure exhibiting three lobes (as shown in Figure S1), and a tetramer structure having four lobes (vide infra).

To further characterize the chemical structures of the D-1 and D-2 dimers, we utilized single-bond resolved nc-AFM imaging in conjunction with DFT calculations. First, we focus on the D-1 structure. From the high-resolution STM image and the corresponding frequency shift AFM image (Figure 2c and 2d,

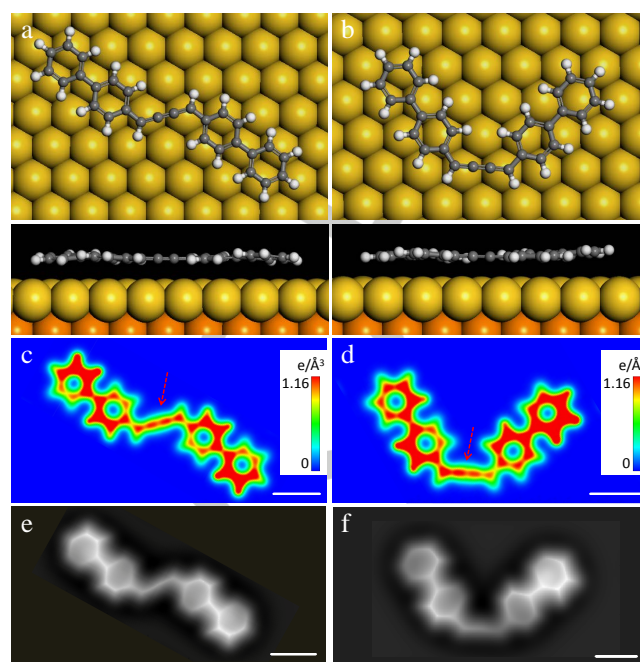


Figure 3. DFT calculations of both cumulenes on Au(111). (a,b) DFT relaxed models of trans- and cis-cumulene products on Au(111). (c,d) Calculated electron density maps of both products, which are cut along a plane parallel to the Au surface above the molecules. The cumulene groups are indicated by red arrows. (e,f) The corresponding simulated constant-height nc-AFM images. Scale bars: 5 Å.

respectively), we identify that the two biphenyl groups are linked together, which confirms the formation of a dimer structure. More importantly, a sharp line with a homogeneous contrast connecting the two biphenyl groups (indicated by a red arrow in Figure 2d) can be clearly discerned. Generally, such features observed in nc-AFM images usually indicate chemical bonds within a molecule.^{40–43} Based on the above analyses, we unambiguously assign this characteristic line feature to the cumulene group having three consecutive C–C double bonds as illustrated in the DFT relaxed model in Figure 2c. In line with D-1, we also imaged D-2 with STM and nc-AFM as shown in Figure 2e and 2f, respectively. We could again clearly resolve the two biphenyl groups linked together by a similar line feature (indicated by a red arrow in Figure 2f). This implies the formation of the cumulene group within the D-2 structure as well (cf. DFT relaxed model in Figure 2e). According to the isomeric configurations of D-1 and D-2, we assign D-1 to the trans-cumulene product, and D-2 to the cis one.

To further verify the formation of the cumulene group, we performed further DFT calculations to construct the relevant structural models of both trans- and cis-cumulene products on Au(111), and calculate the corresponding charge densities. As shown in Figure 3a and 3b, both cumulene products adopt flat-lying geometries on Au(111) except the slight tilt of the benzene rings with respect to each other,^{44,45} and the cumulene group adopts for both cases a linear geometry. The average molecular heights are measured to be 3.1 Å from the DFT models. It has been previously stated that the intramolecular contrast resolved by nc-AFM can be modeled by considering the charge density of the targeted molecules, because the atomic contrast is a consequence of Pauli repulsion.^{47,48} Thus, we calculated the charge density for both cumulene products adsorbed on Au(111), and the cuts along the

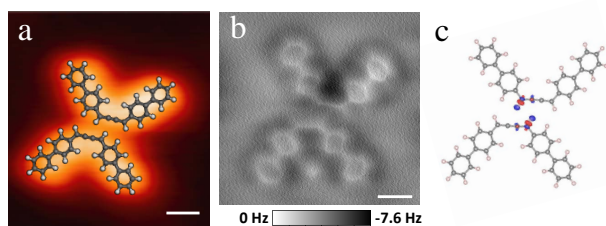


Figure 4. The tetramer structure. a,b High-resolution STM image and the corresponding frequency shift nc-AFM image of a tetramer structure on Au(111). c Charge density difference of the structure showing the intermolecular interactions. The isosurface value is at 0.006 eÅ⁻³. Scale bars: 5 Å.

planes parallel to the surface are shown in Figure 3c and 3d. From these charge density map slices, we can clearly distinguish the biphenyl groups, and more importantly, the cumulene groups exhibit a linear contrast in good agreement with their nc-AFM features. In addition, on the basis of the optimized models, we performed the corresponding nc-AFM simulations by an online modeling software,⁴⁶ which also agree well with our experimental results (cf. Figure 3e and 3f).

Interestingly, we also occasionally observed some tetramers consisting of four lobes on the Au surface. From the combination of high-resolution STM and the nc-AFM measurements (Figure 4a and 4b), we could determine that such a tetramer structure is actually composed of two cis-cumulene dimers. DFT calculations show that these two cis-cumulene dimers are stabilized by a relatively weak intermolecular interaction (CH... π) as reflected by the charge density difference map (Figure 4c), and the binding energy is determined to be 0.18 eV. Note that it is hard to determine from the STM image alone the covalent or non-covalent character between two cis-cumulene dimers within this tetramer structure. Thanks to the simultaneously acquired nc-AFM image, the non-covalent feature between the two dimers could be identified. On the other hand, in comparison with the cis-cumulene dimer structure, it further corroborates the formation of the cumulene group on the surface.

To understand the dehalogenative homocoupling of alkenyl gem-dibromides from a fundamental point of view, we performed extensive calculations to explore the possible reaction pathways from molecular precursors to cumulene products on the Au(111) surface. As shown in Figure 5, we have proposed reasonable reaction pathways of the successive C–Br bond activations followed by subsequent C–C homocouplings on the Au(111) surface (for the calculations, a simplified bBVBP molecule was used, for which the biphenyl group was replaced by a phenyl one). In the first step, a surface stabilized radical is formed by a successive splitting of two C–Br bonds. As shown in Figure 5a, the energy barriers for the successive debrominations on Au(111) are calculated to be 0.91 eV and 0.63 eV, respectively, and the whole dehalogenation reaction is exothermic by 0.52 eV. The energy barrier for the subsequent C–C homocoupling was calculated to be 0.90 eV as depicted in Figure 5b, along with a reaction energy of 1.39 eV. All of these energy barriers are in reasonable agreement with the experimental conditions and could well account for the observed formation of the cumulene products on Au(111).

In conclusion, from the combination of high-resolution UHV-STM and nc-AFM imaging together with DFT calculations, we

successfully fabricated cumulene products on the Au(111) surface by introducing dehalogenative homocoupling reactions of alkenyl

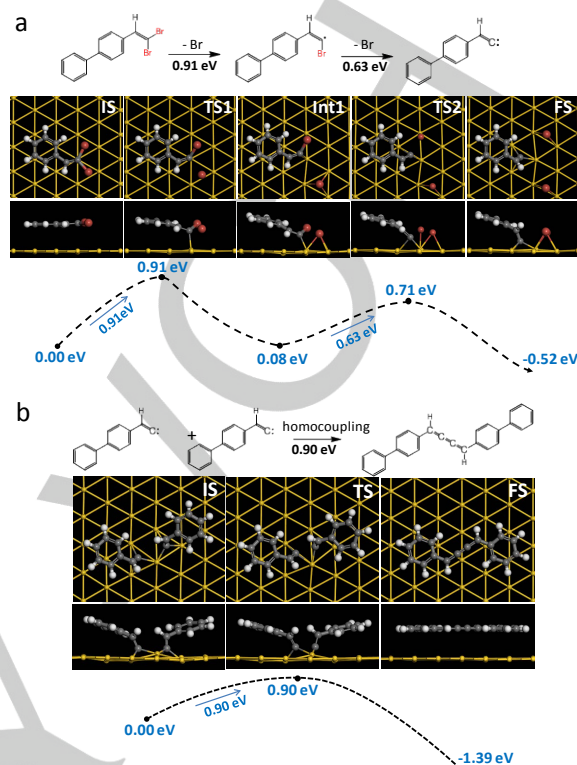


Figure 5. Reaction pathways for the formation of the trans-cumulene. (a) The DFT-calculated reaction pathway for the successive C–Br bond activations of the simplified bBVBP molecule on Au(111). (b) The DFT-calculated reaction pathway from the debrominated intermediates to a cumulene product on Au(111). The structural models of the initial (IS), transition (TS), intermediate (Int) and final states (FS) along the pathways are also shown.

gem-dibromides. Additionally, with the help of DFT calculations we were able to suggest a reasonable reaction pathway for all the steps involved to obtain from the starting monomers to the final cumulene products. It has been recently reported that the elusive aryne has been generated by STM tip excitation on the NaCl/Cu(111) surface and the formed aryne represents a curved cumulene resonance structure.³⁷ Another comparable case is the observation of a cumulene moiety as one of the transient intermediates along an enediyne coupling and cyclization reaction.⁴⁸ Our study sets itself apart from these results since we succeeded for the first time to create linear cumulene structures in a highly controllable fashion. More importantly, it may shed light on the fabrication of increasingly complicated carbon scaffoldings by utilizing the on-surface synthesis strategy.

Acknowledgements

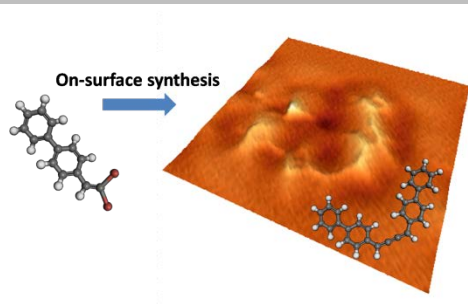
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Keywords: Homocoupling • gem-dibromide • scanning tunneling microscopy • cumulene • surface chemistry

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COMMUNICATION

We have successfully fabricated cumulene products on the Au(111) surface by introducing dehalogenative homocoupling reactions of alkenyl gem-dibromides.



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Liangliang Cai, Honghong Ma,
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